

PARTICULATE CONTAMINATION - TIE-DOWN WITH PARYLENE C

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1.0 Abstract

Entrainment of loose conductive particles within microcircuit packages has been documented as a persistent problem. Occasionally, small, usually microscopic, pieces of metal remain inside hermetically sealed devices despite strenuous efforts to assure cleanliness during manufacturing operations. The problem is of particular concern when hybrid circuits are employed on spacecraft, where stresses encountered during launch can cause previously undetected particles to migrate from innocuous positions to places resulting in electrical shorts. Measures taken, both to test packaged devices for the presence of these particles and to remove them, have included among other means radiographic examination and particle impact noise detection (PIND). Still, small particles can escape notice and non-conducting particles, especially in PIND testing, can cause part rejection.

A positive solution, employed successfully for NASA's Centaur electronics and under evaluation by military agencies, involves a plastic coating, Parylene C. At a thickness of 0.1 mil this material effectively encapsulates and seals (ties down) the offending particles in place, preventing further movement to positions causing electrical malfunction. Additionally, the parylene coats and, thus, insulates all surfaces inside the package so that larger particles, should they manage to break loose, cannot impair electrical performance. Additional process detail will be presented in this review paper.

In contrast to conventional liquid resins, parylene coatings are formed by pyrolysis of high purity di-p-xylylene in a vacuum environment followed by deposition and spontaneous polymerization on cool surfaces. The process resembles vacuum metallizing except that the parylene monomer molecules, while in the gas phase, surround the object to be coated and cause growth of the coating to occur uniformly on all surfaces.

Certain unique features make the parylene system suited for particle tie-down: the ability of the monomer gas to penetrate holes and, thus, coat the inside of microcircuit packages; the ability of the coating to conform precisely to convoluted surfaces including wire bonds and the underside of inverted chip devices; and its ability to cover thoroughly at thicknesses of 0.1 mil and below.

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Compatibility of Parylene C coatings with active and most passive devices has been demonstrated. Documentation of these and the exceptions will be given.

2.0 Introduction

Despite exhaustive measures taken during manufacture, loose conductive particles occasionally remain trapped within hybrid microcircuit packages. Sources of contamination, inspection for it, and ease of removal after various fabrication stages have recently been reviewed¹ as part of an on-going study being made under Air Force sponsorship. While debris generated prior to sealing can generally be observed and removed, solder balls and weld splatter can form during the sealing operation itself. Obviously, contamination of this nature cannot be removed without opening the device. Electrical tests alone do not always reveal the presence of such particles, which can lodge innocuously yet move under spacecraft launch stresses to places where they can cause electrical shorts. As a result, additional testing has become necessary in attempts to prevent contaminated hybrid microcircuits from being installed in critical application areas.

One widely used test regime, particle impact noise detection (PIND), involves vibrating sealed devices while they are acoustically monitored. Costs have been high, due especially to part rejection.² Other approaches are being sought.

One such approach is to immobilize trapped particles within device packages by coating them with plastics or resins. This procedure has become known as "particle tie-down" or "closed package" control. The remainder of this paper describes Parylene C, a vapor deposited plastic, used for this purpose. Silicone resins are also under investigation in the study referenced above.¹

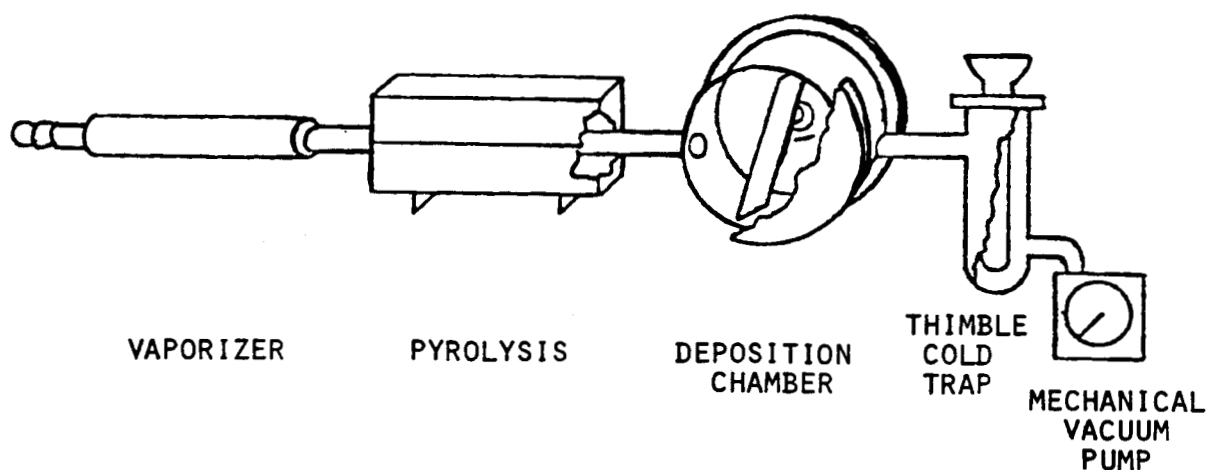
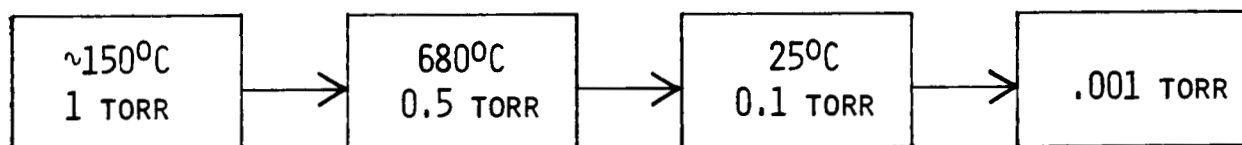
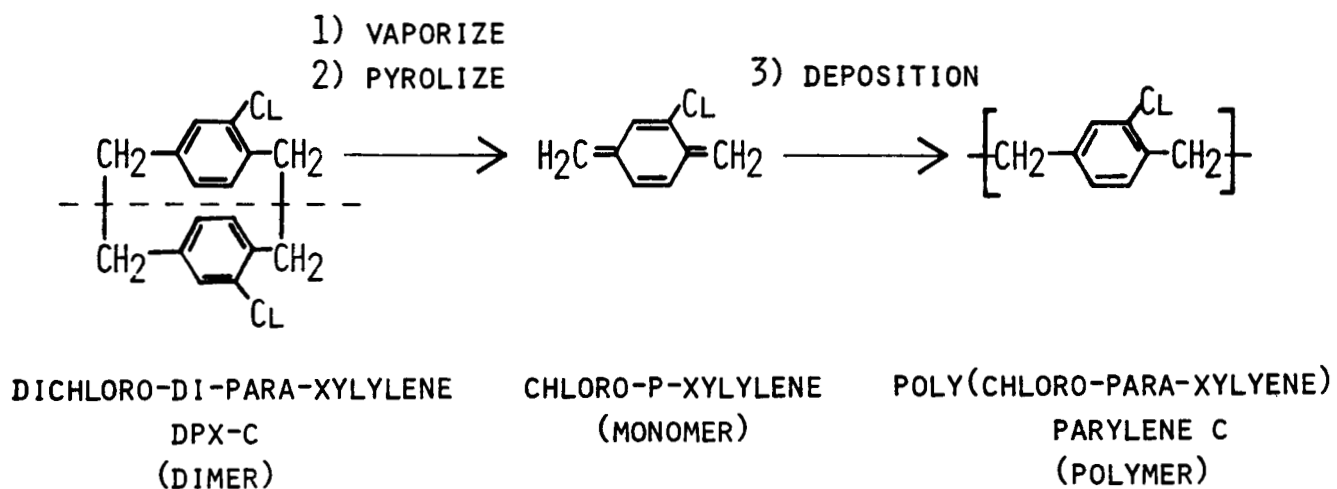
3.0 Parylene C

This family of polymers (Parylene C is one member) is formed by deposition from a low pressure gas directly on the object to be coated. The monomer gas, in turn, is generated from a corresponding high purity solid, called di-para-xylylene; in the case of Parylene C, dichloro-di-para-xylylene or DPX-C. The process is diagrammed in Figure 1.

(¹) Malloy, G. T., Microcircuit Packages Free of Loose Metallic Particles; Quarterly Report IR-S13-6-(1); September, 1977; Air Force Contract F33615-76-C-5273; Hughes Aircraft Company.

(²) Miller, B., Aviation Week and Space Technology, April 11, 1977.

FIGURE 1
PARYLENE DEPOSITION PROCESS



DPX-C, first vaporized at 150°C, 1 mm pressure, passes through a tubular furnace where it is quantitatively pyrolyzed to monomer. The monomer gas enters a chamber containing the substrate to be coated. Substrates remain near room temperature even though pyrolysis occurs at 680°C. As these gas molecules are absorbed on surfaces they polymerize to form a high molecular weight coating. The remainder of the equipment consists of electrical controls, a mechanical vacuum pump, and a cold trap. The trap prevents excess monomer from entering the vacuum pump.

A small parylene coater is pictured in Figure 2. This particular model can accommodate up to 2,000 hybrid circuits, depending mainly upon fixturing. Cycle time is typically four hours.

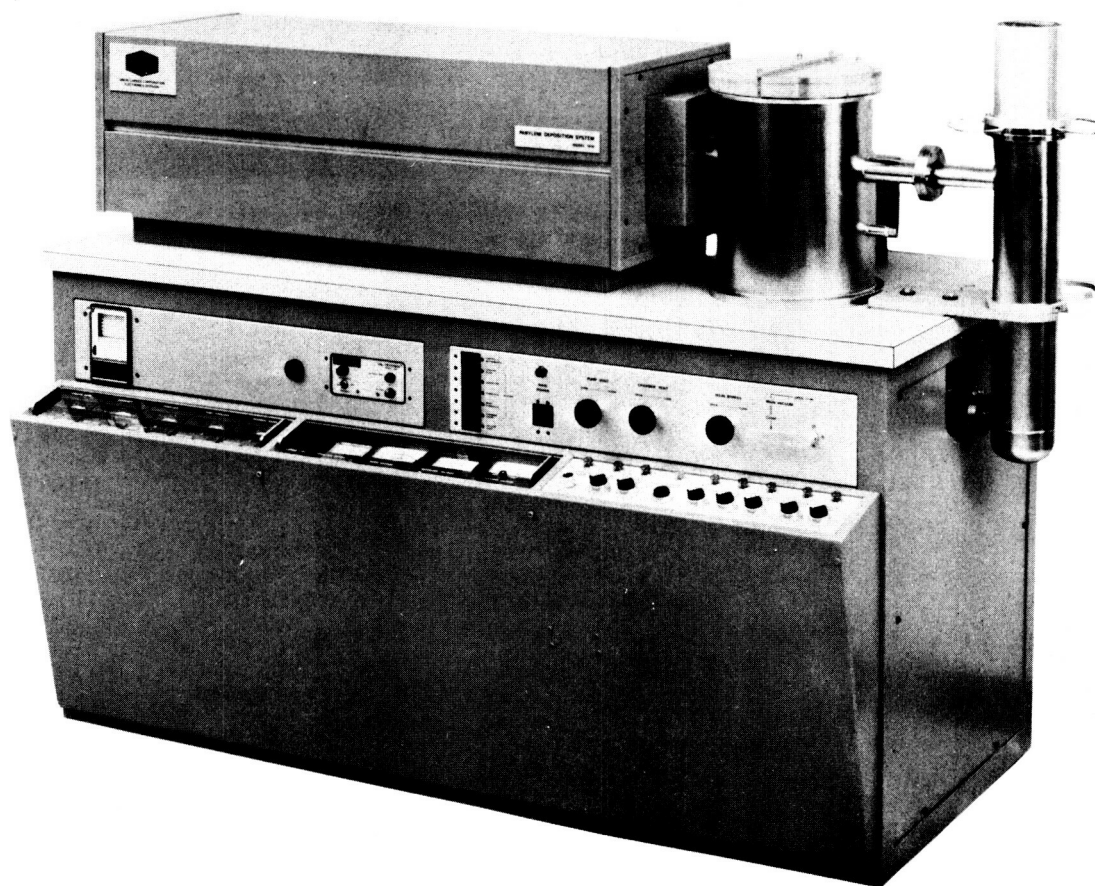


FIGURE 2

PARYLENE COATER-MODEL 10C

Regarding this process, note that spontaneous polymerization occurs on all substrate surfaces without catalysts. Polymerization catalysts are either ionic or free and radical in nature and can adversely affect electrical performance. Furthermore, there is no intermediate liquid state; therefore, post-cure is not required. Curing of liquid resins is usually accompanied by shrinkage with resulting exposure of edges and other stress points. Another feature enhancing the conformality of parylene coatings is the low monomer gas viscosity. Substrates are surrounded and monomer approach takes place at uniform rates from all directions. The process, in other words, has good throwing power and affords protection of areas difficult to access by other means. Conformality is illustrated in Figure 3 which shows a beam lead coated at a thickness of 0.5 mil.



FIGURE 3

BEAM LEAD COATED WITH PARYLENE C
(Courtesy Northrop Electronics)

Parylene N, the parent hydrocarbon of this family of polymers, and parylene D, with two chlorine atoms per aromatic ring, have also been used in selected areas. Detailed property tabulations are given in Appendix A.

In addition to their conformality, the other unique feature of the parylenes is their freedom from pinholes. This is related both to their

spontaneous polymerization without shrinkage and to the physics of the polymerization. Theoretical considerations indicate that chain growth occurs only in the outer 1,000Å surface of the bulk polymer. For application on hybrid circuits, typical thicknesses are 0.15 mils, equivalent to 38,000Å. Thus, growth of a Parylene C coating may be likened to multiple dipping and curing (an accepted means for eliminating pinholes with liquid resins) in which the operations are repeated 38 times.

4.0 Use on Hybrid Circuits

Use of parylene to immobilize particles encountered in hybrid packages was first studied by Hughes Aircraft Company for Goddard Space Flight Center in 1971.³ Subsequently, work at Hughes Aircraft⁴ for Marshall Space Flight Center established the debris holding ability as a function of thickness. Type of contamination and test results are reproduced in Table I.

Vibration and shock testing were conducted both before and after thermal exposure at 125°C for 1,000 hours. From these tests it was concluded that immobilization performance was satisfactory at thicknesses in excess of 0.15 mil and, in most cases, at 0.1 mil or above. In the case of lead shot, it was noted that these particles were substantially larger, 75 mils in diameter, than those encountered in hybrid packages and, being spherical with point contact, were difficult to hold.

Device compatibility was also reported by Hughes and is summarized later in this paper.

Most recently Parylene C has been used to eliminate problems caused by particulate contamination in hybrid microcircuits used on the Centaur launch vehicle. Teledyne Microelectronics Division⁵ working jointly with NASA Lewis Research Center developed tooling for coating hybrid circuits up to 1 1/4 x 1 1/4 inches in size.

At the outset, two coating modes were considered: to coat the devices prior to lidding or to coat through a pretapped hole in the lid, followed by sealing the hole. These alternatives are depicted in Figure 4.

(³) Hughes Aircraft Company, Hybrid Parylene Coating Study, Report Reference No. C-7746/2, August 1972.

(⁴) Oberin, F. W., Development for Application of Parylene Coatings, NAS Contract 8-29940, Final Report, June 1974, NTIS N75-12208, Hughes Aircraft Company.

(⁵) Wylie, D. M., Development of Parylene Passivation for Centaur Hybrid Microcircuits, Final Report NASA CR 135071, Contract NAS 3-19144, August 1976, Teledyne Microelectronics Division.

TABLE I - RESULTS OF SHOCK AND VIBRATION TESTING OF DEBRIS SPECIMENS⁴

Coating Thickness (inches)	Debris Type	Shock (Prior to Thermal Aging)		Shock (After Thermal Aging 1000 Hours at 125°C)			
		Shock	Vibration	Both	N ₂	N ₂ O ₂	N ₂ N ₂ O ₂
0.0001	Lead shot	F	-	-	-	-	-
	Tungsten	P	P	P	P	P	P
	Silicon chips	PF	P	PF	PF	PF	PF
	Gold wire	P	P	P	P	P	P
0.00015	Lead shot	F	-	-	-	-	-
	Tungsten	P	P	P	P	P	P
	Silicon chips	P	P	P	P	P	P
	Gold wire	P	P	P	P	P	P
0.0002	Lead shot	F	-	-	-	-	-
	Tungsten	P	P	P	P	P	P
	Silicon chips	P	P	P	P	P	P
	Gold wire	P	P	P	P	P	P
0.0004	Lead shot	PF*	-	-	-	-	-
	Tungsten	P	P	P	P	P	P
	Silicon chips	P	P	P	P	P	P
	Gold wire	P	P	P	P	P	P
*1 out of 3 failed after six shocks.		P - Passed, PF - Partial Failure, F - Failed					
		N ₂ - 99.9% Nitrogen					
		N ₂ O ₂ - 98.5% N ₂ /1.5% O ₂					

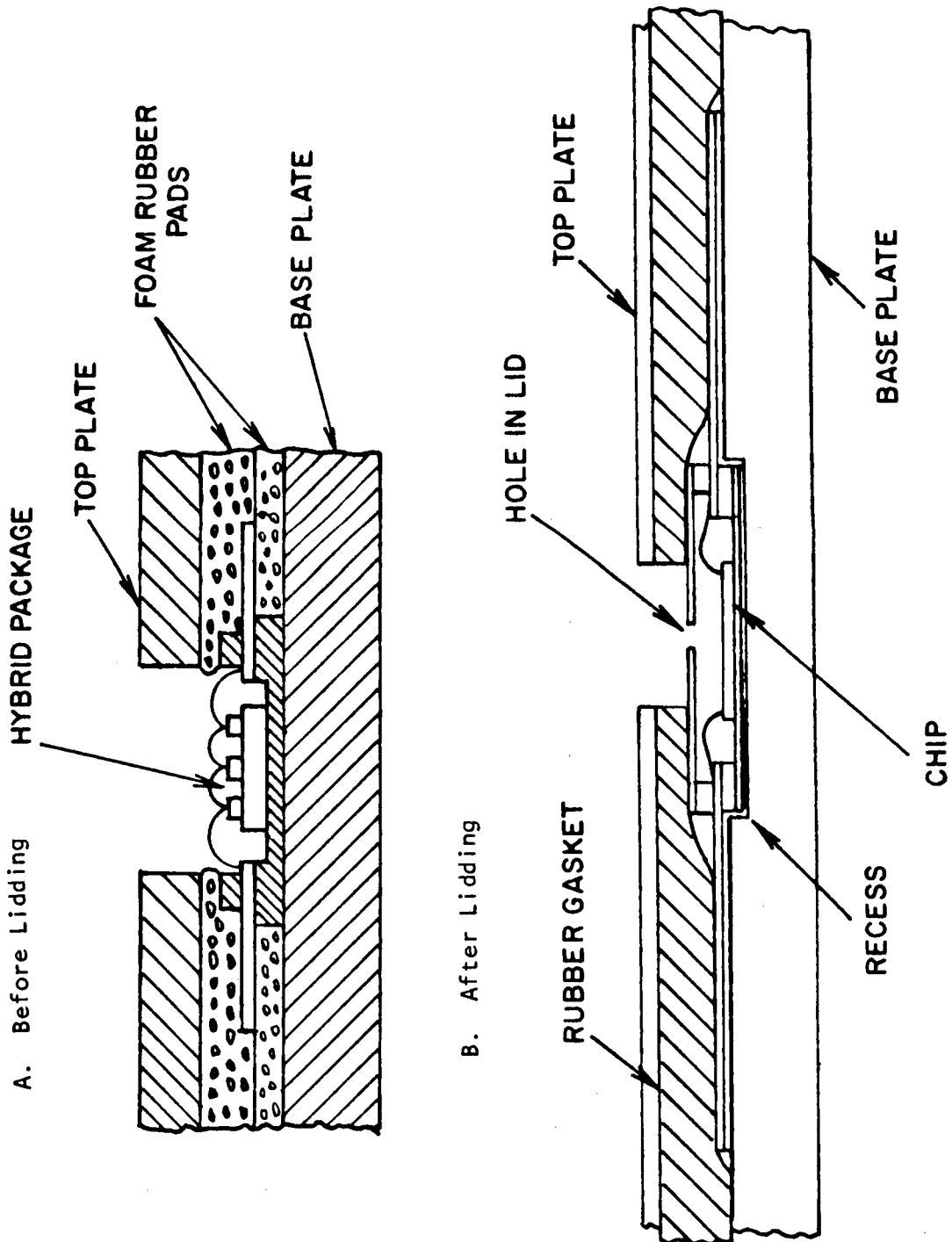
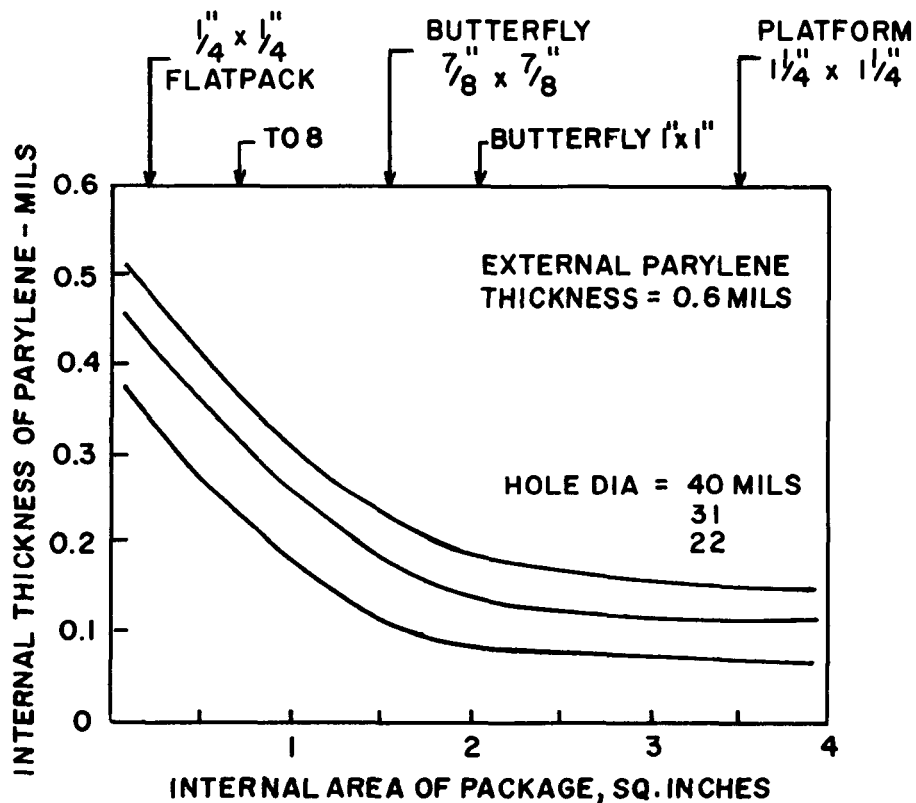


FIGURE 4
ALTERNATIVE MASKING AND HOLDING CONCEPTS

Masking requirements were deemed too extensive for coating prior to lidding; therefore, the through-hole method was chosen despite higher material usage. However, this selection required testing to assure uniform coverage within the package. Five package sizes and three hole diameters were evaluated. Results, given in Figure 5, showed that a centrally located hole, 31 mils diameter, would allow 0.1 mil coating of Parylene C throughout the package, provided the external coating thickness was 0.6 mil.

FIGURE 5
INTERNAL AREA vs INTERNAL PARYLENE
THICKNESS FOR VARIOUS PACKAGES
(COURTESY TELEDYNE MICROELECTRONICS⁵)



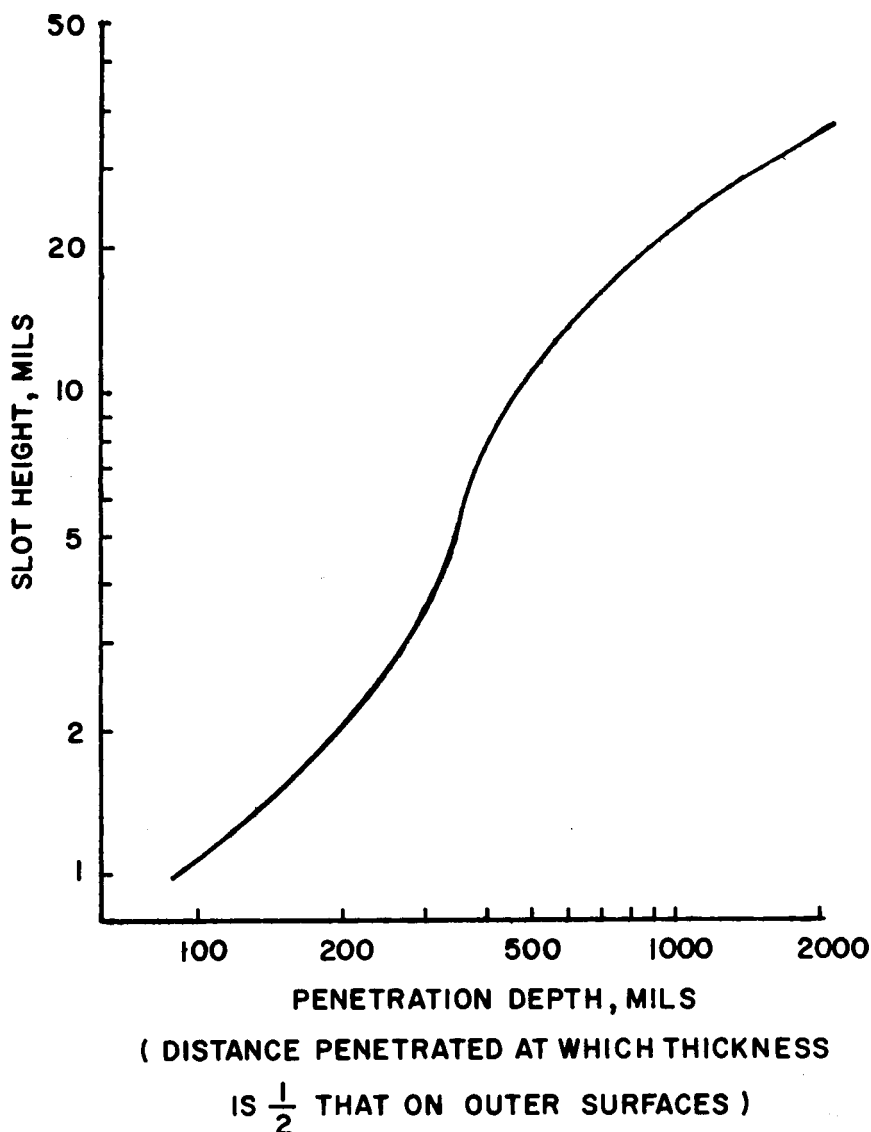
Tooling, in the form of book molds, was constructed which allowed only the hole area of the packages to be exposed. Thus, electrical leads remained uncoated.

Studies of penetration afforded by Parylene C have also been made at Union Carbide. Parallel glass plates, four inches on a side, were spaced at varying distances and sealed on three sides. These were exposed and internal Parylene C film thickness measured as a function of distance into the slot. The results, displayed in Figure 6, show the depth within given slots at which thickness is reduced to one-half that deposited on the external faces.

FIGURE 6

CREVICE PENETRATION OF PARYLENE C

(UNION CARBIDE CORPORATION DATA)



In other words, at an external parylene thickness of 0.5 mil, and for a slot height of 10 mils, penetration would be 400 mils before internal thickness would be lowered to 0.25 mil.

5.0 Component Compatibility

As part of their evaluation of Parylene C for particle tie-down, Hughes Aircraft examined the effect of this product on all types of elements encountered in hybrid circuits. These are listed in Table II with respective measurements and a summary of results.

In general, Parylene C coatings either had no effect upon or benefited the insulation resistance and voltage breakdown performance of conductor patterns, strength of wire bonds, thermal dissipation of wires and beam lead devices, resistance values of thin film resistors, most thick film resistors, and electronic performance of semi-conductors.

The capacitance between conductor patterns was increased over the controls, as expected based on the higher dielectric constant of Parylene C, 3.1 as compared to 1.0 for air.

Thermal aging of coated microwave transmission lines resulted in increased line loss, 22% vs only 7% for the uncoated controls. The reasons are not known. Coated but unaged transmission lines showed improvement in loss characteristics.

Semi-conductor devices for the Hughes study were chosen on the basis of their known sensitivity to surface state conditions. Shifts in electrical behavior, within acceptable limits, were observed for both coated devices and uncoated controls. In general, these were attributed to characteristic sensitivity of the devices and to the test conditions rather than to Parylene C. A possible exception may be the PIN diode. Some PIN diodes treated prior to coating with an adhesion promoter did not recover to acceptable operating limits after HTRB testing.

Low resistivity, unglazed thick film resistors suffered unacceptable resistance increases upon coating with Parylene C, especially after long-term thermal exposure. Performance of coated glazed resistors was acceptable, as was that of unglazed devices with resistance values greater than $1000\Omega/\square$.

While thin film nichrome resistors were unaffected by parylene treatment, it has been reported⁶ that coating with Parylene C speeds reaction with water vapor. Thin film nichrome resistors, when exposed to water under bias, dissolve and open whether coated or uncoated. However, when resistors are fabricated on glazed substrates, coated, and then exposed to

(⁶) Eichel, S., Teledyne Microelectronics, private communication, June, 1977.

TABLE II
EFFECT OF PARYLENE C ON HYBRID CIRCUIT ELEMENTS^a

Element	Measurements	Result Summary
Conductor Patterns	IR, ambient and after 1000 hrs. 150°C. VB, ambient and after 1000 hrs. 150°C. Capacitance ambient and after 1000 hrs. 150°C.	No effect. Coated patterns increased with-stand voltage. Coatings increased capacitance 4-10%.
Wire Bond Strength	Pull strength, as coated and after thermal cycling -65° to +150°C.	As coated specimen strength increased 23-31%; cycled specimens retained strength better than uncoated.
Thermal Dissipation, Wires and Beam Lead Chips	Radiant energy emitted and burn-out current.	Coatings increased emissivity; burn-out current not changed or improved slightly.
Microwave ICs, Microstrip Transmission	Line loss, as coated and after 1000 hrs., 150°C.	As coated had lower loss than controls; aged samples had increased loss.
Thin Film Nichrome Resistors	Resistance, as coated and after 1000 hrs., 150°C.	No effect.
Thick Film Resistors, Glazed	" "	No effect; possible anomaly on 10 Ω □ not caused by parylene.
Thick Film Resistors, Unglazed	" "	No appreciable effect except on 10 Ω □ where aging caused 23% increase.
Semiconductors PIN Diode NPN & PNP Trans. N-Channel MOSFET OP-AMP C-MOS Device	Leakage currents, HTRB	No adverse effects due to coating. Both coated and uncoated PIN diodes showed out-of-limit leakage on HTRB.

humidity, they corrode faster than uncoated controls. It should be noted that an adhesion promoter, standard practice except for through-hole coating, was not used and that non-adherent coatings generally are ineffective in preventing corrosion.

Other than this one instance, it can be stated that no adverse effect of Parylene C on hybrid circuit elements has been reported to Union Carbide.

6.0 Conclusion

Reliability problems encountered in packaging hybrid microcircuits are frequently approached using concepts adopted from the semi-conductor industry. These include careful selection of materials and control of manufacturing operations to assure strength as well as electrical performance, hermetic containers for environmental protection, and extensive screening of finished product to eliminate faulty or marginal units. While extremely low failure rates testify to the success of these methods in most cases, conformal coatings may offer supplementary means or even design alternatives. Cost effectiveness relative to other solutions is the key criterion.

Effectiveness and lower cost have been demonstrated in at least one instance, with the use of Parylene C coatings to resolve a particle contamination problem.

APPENDIX

TABLE I

TYPICAL PHYSICAL AND MECHANICAL PROPERTIES

	Parylene N	Parylene C	Parylene D	Epoxy	Silicone	Urethane
Tensile Strength, psi.	6,500	10,000	11,000	4,000 - 13,000	800 - 1,000	175 - 10,000
Yield Strength, psi.	6,100	8,000	9,000	—	—	—
Elongation to Break, %	30	200	10	3 - 6	100	100 - 1,000
Yield Elongation, %	2.5	2.9	3	—	—	—
Density, g./cm. ³	1.11	1.289	1.418	1.11 - 1.40	1.05 - 1.23	1.10 - 2.5
Coefficient of Friction						
Static	0.25	0.29	0.33	—	—	—
Dynamic	0.25	0.29	0.31	—	—	—
Water Absorption, 24 hours	0.06 (0.029")	0.01 (0.019")	—	0.08 - 0.15	0.12 (7 days)	0.02 - 1.5
Index of Refraction, n _D 23° C.	1.661	1.639	1.669	1.55 - 1.61	1.43	1.50 - 1.60

Data recorded following appropriate ASTM method.

APPENDIX

TABLE II

TYPICAL BARRIER PROPERTIES

Polymer	Gas Permeability cm. ³ -mil/100 in. ² -24 hours-atm. (23°C.)					Moisture Vapor Transmission, g.-mil/100 in. ² -24 hours, 37°C. - 90% RH	
	N ₂	O ₂	CO ₂	H ₂ S	SO ₂	Cl ₂	
Parylene N	7.7	39.2	214	795	1,890	74	1.6
Parylene C	1.0	7.2	7.7	13	11	0.35	0.5
Parylene D	4.5	32	13	1.45	4.75	0.55	0.25
Epoxy	16	66	9	—	—	—	7
Silicone	15	22	45	—	—	—	290
Urethane	3	3	8	—	—	—	12

Data recorded following appropriate ASTM method.

APPENDIX

TABLE III

TYPICAL ELECTRICAL PROPERTIES

	Parylene N	Parylene C	Parylene D	Epoxy	Silicone	Urethane
Dielectric Strength, Short Time, volts/mil at 1 mil	7,000	5,600	5,500	2,300	2,000	3,500
Volume Resistivity, 23°C., 50% RH, ohm-cm.	1 x 10 ¹⁷	6 x 10 ¹⁶	2 x 10 ¹⁶	1 x 10 ¹⁴	1 x 10 ¹⁵	2 x 10 ¹⁵
Surface Resistivity, 23°C., 50% RH, ohms	10 ¹³	10 ¹⁴	5 x 10 ¹⁶	5 x 10 ¹³	3 x 10 ¹³	6 x 10 ¹⁴
Dielectric Constant						
60 Hz	2.65	3.15	2.84	4.2	2.6	3.5
10 ³ Hz	2.65	3.10	2.82	3.9	2.6	3.4
10 ⁶ Hz	2.65	2.95	2.80	3.4	2.6	3.2
Dissipation Factor						
60 Hz	0.0002	0.020	0.004	0.03	0.0005	0.01
10 ³ Hz	0.0002	0.019	0.003	0.03	0.0004	0.01
10 ⁶ Hz	0.0006	0.013	0.002	0.04	0.0008	0.01

Data recorded following appropriate ASTM method.

APPENDIX

TABLE IV

TYPICAL THERMAL PROPERTIES

	Parylene N	Parylene C	Parylene D	Epoxy	Silicone	Urethane
Melting or Heat Distortion Temperature, °C.	405	280	>350	up to 220	up to 300	170
Linear Coefficient of Expansion, ($10^{-5}/^{\circ}\text{C.}$)	3.5	6.9	—	4.5 - 6.5	25 - 30	10 - 20
Thermal Conductivity, (10^{-4} cal./sec./cm. 2 ·°C./cm.)	~3	—	—	4 - 5	3.5 - 7.5	5

Data recorded following appropriate ASTM method.